Ultra-low accelerating voltage observation of a thin carbon membrane with a deceleration optics system scanning electron microscope

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Secondary electron (SE) images of the surface of a thin carbon microgrid (CM) firmly covering a copper plate substrate were investigated using a deceleration optics system SEM. The CM was prepared by carbonization of a formvar microgrid used for the transmission electron microscope, 40 nm in thickness, at 700 °C in a N\textsubscript{2} atmosphere, placed on a smooth flat copper plate substrate. The CM is regarded to be composed of a low conductivity material. The estimated thickness was 40 nm. Observation was conducted with the electron probe accelerated at voltages in the range 0.1-30.0 kV. Characteristic SE images of the CM-covered copper plate substrate as a function of accelerating voltage \(V_0\), i.e. probe electron energy \(E_0\), were obtained. The clearest images of the CM surface were observed with \(E_0\) in the range 0.1-1.0 keV. The variation of the SE image versus \(E_0\) was interpreted with the aid of a Monte Carlo electron trajectory simulation for the CM-covered copper plate substrate.

**KEYWORDS**: Deceleration optics system SEM, Ultra-low accelerating voltage observation, Carbon membrane, Monte Carlo electron trajectory simulation, Interaction volume

1. Introduction

The scanning electron microscope (SEM) is an excellent device for characterization of the surface texture of carbon materials. Various imaging techniques are available for observation of texture with the SEM, and among them the most useful technique is scanning electron microscopy which, for imaging, utilizes the secondary electrons (SEs), released valence electrons, released from the specimen atoms near the specimen surface with irradiation of a finely focused electron beam. The electron beam is called an electron probe.

For carbon materials, thin sheet materials such as carbon and graphite membranes are becoming common in practical use\textsuperscript{1)} and the observation of the surface texture of such materials using SE images is necessary on some occasions. For observation, the relationship between the specimen thickness and maximum penetration depth of the probe electrons as a function of accelerating voltage of probe electrons must be known. An important case is that where the sheet material is thinner than the maximum penetration depth. A thin sheet specimen is usually prepared by covering a flat surface of a substrate with the specimen material. For such a specimen, when the maximum penetration depth is deeper than the thickness of the sheet specimen, the resultant SE images can be affected considerably by the substrate material. The present study concerns an SE image observation of a thin sheet material firmly covered on a flat copper plate substrate. As the sheet material, a carbon membrane specimen of 40 nm in thickness was considered.

The surface texture of the thin sheet specimen, as SE images, can be observed when the maximum penetration depth of the probe electrons is less than the specimen thickness. The maximum penetration depth can be estimated to 32 nm at an accelerating voltage \(V_0\) of 1.0 kV and 48 nm at 1.5 kV for the present carbon membrane through Monte Carlo electron trajectory simulation, as will be described later. This simulation result suggests that when \(V_0\) exceeds marginally 1.0 kV, some of the probe electrons pass through the sheet and excite substrate atoms. The effect of such probe electrons should be considered for qualification of the SE image as a function of \(V_0\). For the investigation, the minimum value of \(V_0\) should be of the order of 0.1 kV at the least. On the other hand, estimation of the maximum escape depth of the SE is important even for the order of magnitude. According to Seiler, with a mean escape depth of the SE \(\lambda\), the maximum es-

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cape depth of the SE can be assumed to be as large as \( 5 \lambda \). \( \lambda \) depends on the specimen material, about 0.1-0.5 nm for metals and 10-20 nm for insulators. Currently, however, SE image observations for carbon materials at accelerating voltages as low as 0.1 kV have not been reported, since for accelerating voltages below 1 kV the aberration of the probe electrons focused on the specimen surface becomes larger, with lower accelerating voltages.

Recently, a deceleration optics system SEM operating from extremely low accelerating voltages of probe electrons down to 0.1 kV has been developed. It is based on the deceleration, i.e. retardation, of low aberration probe electrons just before entering the specimen. The present study concerns SE image observation using this kind of SEM for a carbon membrane specimen of 40 nm in thickness covering a copper plate substrate. Firstly the dependence of the SE image on the accelerating voltage of the electron probe, i.e. probe electron energy, was obtained and then the interpretation of the SE image is discussed through the related Monte Carlo electron trajectory simulation.

2. SEM Observation and SE Images for the Carbon Membrane

2.1 Preparation of the carbon membrane

A formvar microgrid for a transmission electron microscope (TEM) of 40 nm in thickness was placed on a smooth flat copper plate substrate and was carbonized by heating to 700 °C in a N₂ atmosphere. The residence time for carbonization heating at the top temperature was 60 min. After carbonization, the carbonized formvar microgrid became firmly attached to the copper plate. The carbon microgrid, i.e. the carbon membrane, is abbreviated hereafter as CM. The thickness of the CM \( z_{CM} \) was evaluated to about 40 nm by TEM image observation of the CM. Since the CM is a carbon material carbonized at 700 °C, its room temperature conductivity could be regarded as a value of the order of \( 10^{-6} \text{ S m}^{-1} \).

2.2 SE image observation

The SEM used for observation was a Hitachi SU8200 FE SEM. The SU8200 is an in-lens SEM, providing a space at the tip of the objective lens for the specimen to reduce the objective lens aberration. The SU8200 provides also a deceleration system for low aberration probe electrons. “Low aberration probe electrons” means here probe electrons accelerated at accelerating voltages of above 5 kV. Deceleration of the probe electrons is achieved with application of a negative voltage \( V_{OS} \) between the tip of the objective lens and specimen to reduce \( V_0 \) down to 0.1-1.0 kV in steps. The deceleration system is schematically depicted in Fig. 1. In Fig. 1, \( V_0 \), \( V_{OS} \) and \( V_0' \) are related to \( V_0' = V_0 + V_{OS} \), with \( V_{OS} < 0 \). Numerically, the low aberration probe electrons with \( V_0' = 5 \) kV focusing on the specimen surface can be decelerated to 0.2 kV by application of \( V_{OS} \) of −4.8 kV, for example.

Three detectors which detect SEs, backscattered electrons (BES) and low-loss back scattered electrons, respectively, for their respective imaging types, are placed at different positions in the SEM. Two of the three are set close to the lower and upper positions of the objective lens; the upper detector detects electrons with energies below 50 eV, mainly SEs, while the lower detector detects electrons with energies above 50 eV. The latter electrons are the probe electrons scattered out from the specimen surface and are referred to as BES. The third detector is set close to the filter provided above the objective lens to allow passage of low-loss BESs. Only the upper detector concerns the present study.

The electron probe was irradiated normal to the CM surface. SE images were taken using the upper detector as a function of \( V_0 \), with \( V_0 \) set at 0.1, 0.2, 0.3, 0.5, 1.0, 2.0, 3.0, 5.0, 7.0, 10.0, 15.0, 20.0 and 30 kV, while keeping the same visual field and magnification. Images with \( V_0 \) at 0.1 and up to 1.0 kV were obtained using the deceleration system.

2.3 Observed SE images

The SE images obtained are depicted in Fig. 2. In each SE image, a left right arrow is inset at the bottom right of the image as a marker for magnification, the length of the arrow being 2.00 µm. At accelerating voltages of 1.0 kV and below, the voltage value in the marginal note of the image is indicated by the accelerating voltage followed by -D, to signify use of the deceleration system, 1.0 kV-D for example. At voltages of 1.0 kV and above the note signifies the accelerating voltage only. The SE image obtained depends strongly on the accelerating voltage and has the following features.

1) The SE images with the electron probes accelerated at \( V_0 \) in the range 0.1-1.0 kV using the deceleration system are similar. In each SE image, the CM shows large open holes created at the preparation stage of the raw formvar microgrid. The surface texture of the CM can be seen clearly. In each hole, the granular surface texture of the copper plate can be seen and, extends over the whole surface.

2) The SE image taken at \( V_0 = 1.0 \) kV without use of the deceleration system is similar to that obtained with use of the deceleration system.
3) The image of the CM at $V_0 = 2.0$ kV changes significantly from the image taken at $V_0 = 1.0$ kV. Circumference edges surrounding the open holes brighten up the enclosing boundary of each hole as a curve having a width. The width decreases with increasing $V_0$ up to 7.0 kV and then levels off.

4) In the images of the CM, an exceedingly faint image of the surface texture of the copper plate appears at $V_0$ of 2.0 kV, and the CM image starts to transparentize at $V_0$ of 3.0 kV with the appearance of a granular texture to the copper plate surface under the CM. The images of the copper plate surface under the CM taken at $V_0$ values of 5 and 7 kV are similar.

5) The transparency of the CM progresses with increasing $V_0$ up to 7 kV.

3. Interpretation of SE Images of the CM-covered Copper Plate Substrate

The probe electrons irradiating the specimen diffuse into the specimen and interact with the specimen atoms both inelastically and elastically. To excite the specimen atoms and to release the SEs results in an inelastic scattering. Only the SEs released from the specimen atoms in the region near the specimen surface can escape because of their low energy, since the energy range for SE excitation is below 50 eV. This low energy range can be confirmed by the disappearance of the SE image when a positive voltage is applied between the specimen and the ground below 50 V. The maximum depth in the region from the specimen surface is the maximum escape depth of the SE. The maximum escape depth of the SE in the CM can be estimated to...
the order of 5 nm.

Elastic scattering is scattering without energy loss, which causes a change in direction of the trajectory of the diffusing probe electrons. In the course of the diffusion, some of the probe electrons suffer inelastic and elastic scatterings frequently with different specimen atoms; finally they lose almost all of their energy and are absorbed by the specimen. Other probe electrons are scattered out from the specimen surface with the final elastic scattering. These scattered out probe electrons are the BEs. The maximum penetration depth of the probe electron from the specimen surface can be related to the point where the probe electron changes its direction of motion.

BEs, when they leave the specimen, excite the specimen atoms near the specimen surface and release SEs. These SEs are designated as extra SEs (ExSEs). Energy of ExSEs is similar to that of SEs, and ExSEs are detected by the upper detector of the SU8200. Since ExSEs include the information of the deep inside of the specimen, i.e. the Cu plate substrate, they disturb the pure SE image at the specimen surface.

3.1 Escape depth of secondary electrons

As described in Introduction, the maximum escape depth of SEs can be evaluated to \( 5 \lambda \), \( \lambda \) being defined below. The escape probability of the SE from the specimen is known to decrease exponentially with depth

\[
p \propto \exp(-z/\lambda) \quad \text{..........................................................(1)}
\]

here \( p \) is the escape probability of the SE, \( z \) is the depth below the specimen surface where the SE is excited and \( \lambda \) is the depth for \( p=1/e \) (e: the base of the natural logarithm). Since \( \lambda \) values are regarded 0.1-0.5 nm for metals and 10-20 nm for insulators and if the conductivity of the insulator is taken to be of the order of \( 10^{-10} \text{ S m}^{-1} \), the \( \lambda \) value for the CM could be considered to be of the order of 1 nm because of the conductivity value being of the order of \( 10^{-8} \text{ S m}^{-1} \).

3.2 Monte Carlo depiction of the interaction volume for the CM-covered copper plate substrate

3.2.1 Simulation procedure

When inelastic and elastic interactions occur they distribute the probe electron trajectories over a three dimensional volume. This volume is called the interaction volume. The Monte Carlo electron trajectory simulation is a simulation to infer the interaction volume. The simulation was conducted here for the CM-covered copper plate substrate modeled as two component materials using open source software constructed by Kanda. The simulation was also performed for a large size specimen of formvar carbonized at 700 \( ^\circ \text{C} \) having the same density as that of the CM, 2,000 g cm\(^{-3}\) and a copper plate with a density of 8.993 g cm\(^{-3}\). The large size formvar carbon is abbreviated hereafter as FVC. The reason for the density of 2,000 g cm\(^{-3}\) being assumed for CM and FVC is due to the crystallite unit cell density for these materials.

Though an X-ray diffraction study for the SE was not conducted in the present study, the unit cell volume can be estimated from the unit cell volume data of Kapton carbons heat-treated between 900 and 2200 \( ^\circ \text{C} \) with steps of 100 \( ^\circ \text{C} \). The lattice constants \( a_0 \) and \( c_0 \) of each heat-treated Kapton carbon were obtained differently, but their unit cell volumes can be evaluated to be the same values of \((3.60 \pm 0.03) \times 10^{-23} \text{ cm}^3\). This unit cell volume gives the same density of 2.21 \( \pm 0.02 \text{ g cm}^{-3}\). For the CM crystallite, the density can be assumed to be this value. Since in the SE images for surface texture of the CM with accelerating voltages below 1.0 kV in Fig. 2 no appreciable pores can be seen, nanometric pores due to curved connections of the crystallites are supposed. If the texture of the CM contains 10% nanometric scale pores by volume, the density of the CM can be assumed to be 2.00 \( \pm 0.02 \text{ g cm}^{-3}\). For the simulation, the density of the CM was set with 4 significant figures.

During the simulation, the thickness of the CM was set at 40 nm, and the FVC was taken as the reference material for the CM. The simulation was made with an irradiation of 500 electrons having a given energy \( E_0 \) to the specimen surface, here \( E_0 = Ev_0 \) in eV units and \( e \) is the proton charge. The \( E_0 \) values were set at 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 5.0, 7.5, 10.0 and 15.0 kV. For a given \( E_0 \), simulations were made 6 times. Preliminary tests of the simulations with densities of 2.000 and 1.800 g cm\(^{-3}\) set were made for several \( E_0 \) values and comparable results were obtained.

3.2.2 Simulation results and interaction hemisphere

The simulated interaction volumes for CM-covered copper plate substrate are depicted in Fig. 3. Each simulation figure in Fig. 3 shows a projection of the electron trajectories on a vertical plane including the irradiation direction of the probe electrons taken as the Z-axis. Fig. 4 demonstrates interaction volumes for FVC and copper plate with \( E_0 \) at 0.5 and 2.0 kV, respectively.

Goldstein et al. considered the Monte Carlo depiction of the interaction volume as a hemisphere constructed with a radius whose origin is the entry point of the probe electrons at the specimen surface and containing a specified fraction of electron trajectories. In the present study, the interaction volume is defined as an interaction hemisphere containing the electron trajectories within the volume. Trajectories can be represented by their terminal points. There are a great many numbers of trajectories in the hemisphere, in reality, and the terminal points can be assumed to be distributed homogeneously in the hemisphere. The radius of the interaction hemisphere is equivalent to the maximum penetration depth of the electron probe into the specimen at a given \( E_0 \). Fig. 5 depicts the radii of the interaction hemispheres \( r_{\text{CM,Cu}, \text{FVC}} \) and \( r_{\text{Cu,plate}} \) for CM-covered copper plate substrate, FVC and copper plate, respectively, as a function of \( E_0 \). So, the penetration depths at the low \( E_0 \) region, i.e. low \( V_0 \) region, become 32 nm at \( V_0 \) of 1.0 kV and 48 nm at 1.5 kV. The \( r_{\text{CM,Cu}} \) values in the low \( E_0 \) region, \( E_0 \) below 1.0 keV, are similar to those for FVC,
while the values in the high $E_0$ region are similar to those for copper plate. Low $E_0$ behaviors in Fig. 5 provide a condition of $E_0$ for a clear surface image of the CM.

The best fit curve for the $E_0$ vs. $r_{CM, Cu}$ relationship in Fig. 5 can be obtained as depicted in Fig. 6, where $[r_{CM, Cu}]_0$ is the fitted value for the $r_{CM, Cu}$. The $E_0$ value for the $[r_{CM, Cu}]_0$ of 80 nm is obtained as 2.19 keV. Probe electrons with energies of 2.2 keV and above are backscattered from the copper plate, excite specimen carbon atoms in the CM and release ExSEs. These ExSEs involve the information of the image of the surface texture of the copper plate and disturb the SE image of the surface texture of the CM at $E_0$ above 2.2 keV. This means that when $E_0$ is increased from a low value, a clear SE image of the surface texture is observed, with increasing $E_0$ similar SE images are obtained and the image suddenly changes at $E_0$ of 2.2 keV. Actually, the image change in Fig. 2 occurs at 2.0 keV. The $E_0$ value of 2.0 keV gives a $r_{CM, Cu}$ value of 73 nm in Fig. 5, and the CM thickness of 40 nm could be an overestimate. Hereafter, the $2z_{CM}$ value is regarded as 73 nm and the $E_0$ value of 2.2 keV above is replaced with 2.0 keV.

### 3.2.3 Backscatter coefficient

In each simulation, the backscatter coefficient $\eta$ defined by $\eta = n_{BE}/n_p$ was obtained for FVC, CM-covered copper plate substrate and copper plate itself as well as the probe electron trajectories, where $n_{BE}$ and $n_p$ are the numbers of backscattered electrons and probe electrons, respectively$^{5,6}$. $\eta$ is known to depend on the energy of the electron probe.

The values of $\eta$ obtained for the CM-covered copper plate substrate, FVC and copper plate are plotted as a function of $E_0$ in Fig. 7. $\eta$ for $E_0$ at 2.0 keV and below shows a variation close to that of FVC, while for $E_0$ at 7.5 keV and above $\eta$ changes as it changes in the cop-
Large size formvar carbon (FVC)

Copper plate

Fig. 4 Monte Carlo electron trajectory simulations of the interaction volume in FVC and copper plate for $E_0 = 0.5$ and $2.0$ keV.

Fig. 5 Interaction hemisphere radius for CM-covered copper plate, $r_{CM,Cu}$ (○), FVC, $r_{FVC}$ (■), and copper plate, $r_{Cu\ plate}$ (▲), as a function of $E_0$.

Fig. 6 $E_0$ vs. $[r_{CM,Cu}]_{fit}$ for CM-covered copper plate.

Fig. 7 Backscatter coefficient $\eta$ for CM-covered copper plate, $\eta_{CM,Cu}$ (○), FVC, $\eta_{FVC}$ (■), and copper plate, $\eta_{Cu\ plate}$ (▲), obtained from Monte Carlo electron trajectory simulations as a function of $E_0$.

\[
\eta V_{CM,Cu} = n_{BE} / (n_p / V_{CM,Cu})
\]  

(2)

per plate. $\eta V_{CM,Cu}$, $V_{CM,Cu}$ being the volume of the interaction hemisphere in the CM-covered copper plate substrate, can be written

here $n_p/V_{CM,Cu}$ is the number of probe electrons per interaction hemisphere volume, i.e. the probe electrons per unit volume. For the FE SEM $n_p/V_{CM,Cu}$ can be supposed to be a constant. Using Eq. (2), the $n_{BE}$ value normalized to the value at $E_0 = 15$ keV, $n_{BE}[n_{BE}]_{15}=\eta V_{CM,Cu}/[\eta V_{CM,Cu}]_{15}$, is plotted as a function of $E_0$ in Fig. 8. In Fig. 8, the $E_0$ values should be limited to those above 2.0 keV, though the plots are made at 0.5 keV and above. Fig. 8 demonstrates the increasing nature of BEs against $E_0$. 
3.3 Schematic depictions for escapes of SEs, ExSEs and BSs

The feature of the escapes of SEs, ExSEs and BSs can be considered according to the size of $r_{\text{CM}, \text{Cu}}$.

3.3.1 $r_{\text{CM}, \text{Cu}} \leq z_{\text{CM}}$

The interaction hemisphere is defined in the CM and the volume $V_{\text{CM}}$ is expressed as $V_{\text{CM}} = \left(\frac{2\pi}{3}\right) r_{\text{CM}, \text{Cu}}^3$. The SEs escape from the depths within the maximum escape depth of SE, $D_{\text{SE}}$, which are of the order of 5 nm. BSs are scattered from the depths within the maximum escape depth of BE, $(1/2)r_{\text{CM}, \text{Cu}}$, and they excite the carbon atoms in the CM and release ExSEs when they leave the specimen. The released ExSEs escape from $D_{\text{SE}}$ and are detected with the upper detector of the SU8200. ExSEs detected are denoted hereafter as $(\text{ExSE})_{\text{CM}}$.

The features of the escape of SEs, $(\text{ExSE})_{\text{CM}}$s and BEs are depicted in Fig. 9(a) and (b), where SEs, $(\text{ExSE})_{\text{CM}}$s and BEs are indicated by arrows from each maximum escape depth of SEs, $(\text{ExSE})_{\text{CM}}$s and BEs. SEs and $(\text{ExSE})_{\text{CM}}$s are detected by the upper detector for SE image forming. As depicted in Fig. 9(b), since $D_{\text{SE}}$ is usually shallower than $(1/2)r_{\text{CM}, \text{Cu}}$ ($(\text{ExSE})_{\text{CM}}$s give the information within the depth $(1/2)r_{\text{CM}, \text{Cu}}$, but disturb the exact SE image a bit. However, when $E_0$ is quite low as drawn in Fig. 9(a), $D_{\text{SE}}$= $(1/2)r_{\text{CM}, \text{Cu}}$ and SEs and $(\text{ExSE})_{\text{CM}}$s give similar information, exact SE images can be obtained. BSs are detected by the lower detector of the SU8200.

3.3.2 $z_{\text{CM}} < r_{\text{CM}, \text{Cu}} \leq 2z_{\text{CM}}$

The interaction hemisphere in this case extends over the CM and copper plate substrate. As depicted in Fig. 9(c), the interaction hemisphere cut off a CM disk $\text{EFF/E}^\prime$ with a volume $\mathcal{V}_{\text{CM}}$ from the CM and a copper disk with a volume $\mathcal{V}(\text{Cu}; z_{\text{CM}}, (1/2)r_{\text{CM}, \text{Cu}})$ from the copper plate substrate. $\mathcal{V}_{\text{CM}}$ and $\mathcal{V}(\text{Cu}; z_{\text{CM}}, (1/2)r_{\text{CM}, \text{Cu}})$ are evaluated to

$$\mathcal{V}_{\text{CM}} = \left(\frac{\pi}{3}\right) (3r_{\text{CM}, \text{Cu}}^2 z_{\text{CM}} - z_{\text{CM}}^3) \quad \text{with} \quad z_{\text{CM}} \geq z > 0$$

The maximum escape depth of BE in this case is $(1/2)r_{\text{CM}, \text{Cu}}$.

As depicted in Fig. 9(c), SEs escape from the depth $D_{\text{SE}}$, BSs escape from any depth within the CM and they let ExSEs, denoted...
as (ExSE)_{CM}, escape from a depth within $D_{SE}$ when they leave the specimen. Resolution of the SE images is reduced by (ExSE)_{CM}, being more than the case $r_{CM,Cu} \leq z_{CM}$. BEs are detected by the lower detector of the SU8200.

### 3.3.3 $r_{CM,Cu} \geq 2z_{CM}$

The interaction hemispheres with a radius larger than $2z_{CM}$ can be sectioned into three parallel disks stacked as depicted in Fig. 10; a CM disk with a volume $V_{CM}$, cut off by the upper half of the interaction hemisphere corresponding to a CM disk $GHH’G’$ in Fig. 9(d), a copper disk with a volume $V(Cu; z_{CM}, (1/2)r_{CM,Cu})$ cut off by the lower half of the interaction hemisphere corresponding to a disk $JKJ’$ in Fig. 9(d). Fig. 9(d) depicts escapes of SEs, (ExSE)_{CM}, (ExSE)_{Cu,pl} and BSs. Though BSs are indicated with an arrow with a mark BS, they consist of the BSs backscattered from any depth within the CM, i.e. within the depth $z_{CM}$, denoted as (BS)_{CM}. From the CM, similar to the case $z_{CM} < r_{CM,Cu} \leq 2z_{CM}$, SEs and (ExSE)_{CM} escape from the specimen and are detected with the upper detector for SE image forming. From the volume $V(Cu; z_{CM}, (1/2)r_{CM,Cu})$, BSs denoted as (BS)_{Cu,pl} are backscattered and they let ExSEs denoted as (ExSE)_{Cu,pl} escape from the depth $D_{SE}$. (ExSE)_{Cu,pl} are detected by the upper detector but they have considerable information on the texture of the copper plate substrate and could degrade exact SE images significantly. The volume $V(Cu; z_{CM}, (1/2)r_{CM,Cu})$ is inactive for the backscattering of probe electrons. Volumes $V_{CM}$, $V(Cu; z_{CM}, (1/2)r_{CM,Cu})$ and $V(Cu; r_{CM,Cu}/2, r_{CM,Cu})$ can be evaluated to

$$V_{CM} = \frac{\pi}{3}(3r_{CM,Cu}z_{CM} - z_{CM}^3) \quad \text{with} \quad z_{CM} \geq z > 0,$$

$$V(Cu; z_{CM}, r_{CM,Cu}/2) = \frac{\pi}{24}(11r_{CM,Cu}^3 - 24r_{CM,Cu}z_{CM} + 8z_{CM}^3) \quad \text{with} \quad (1/2)r_{CM,Cu} \geq z > z_{CM}$$

and

$$V(Cu; r_{CM,Cu}/2, r_{CM,Cu}) = \frac{5}{24}r_{CM,Cu}^3 \quad \text{with} \quad r_{CM,Cu} \geq z > (1/2)r_{CM,Cu}$$

In the CM-covered copper plate substrate in reality, each interaction hemisphere contains exceedingly large number of electron trajectories. It can be assumed that the volumes $V_{CM}$ and $V(Cu; z_{CM}, (1/2)r_{CM,Cu})$ reflect the number of (SE)_{CM}, $n_{SE,CM}$, and the number of (BE)_{CM}, $n_{BE,CM}$. The ratio $V(Cu; z_{CM}, r_{CM,Cu}/2)/V_{CM}$ gives the ratio $n_{BE,CM}/n_{SE,CM}$. Fig. 11 plots $V_{CM}$ and $V(Cu; z_{CM}, (1/2)r_{CM,Cu})$ (a) and $V(Cu; z_{CM}, r_{CM,Cu}/2)/V_{CM}$ (b) as a function of $E_{0}$. Since $n_{BE}$ approximates the number of ExSE $n_{ExSE}$. Fig. 11 suggests that $n_{ExSE}$ dominates at $E_{0}$ above 5.0 keV. For $E_{0}$ above 5.0 keV, the SE images are almost only due to $n_{ExSE}$ and the texture image of the copper plate substrate becomes much clearer with further increase of $E_{0}$.

### 3.4 Edge effect at circumference edges surrounding holes of CM

As described in Section 2.3, a peculiar feature of the images in Fig. 2 is that an edge effect for the circumference edges surrounding the holes is related to $E_{0}$, accordingly $E_{0}$. The edge effect starts to appear suddenly at $E_{0}$ of 2.0 keV, with brightening up along the curve showing the enclosing boundary of each hole. The curve has a width and the width becomes narrower with increasing $E_{0}$ up to 7 keV with a sharp edge image and levels off. This type of the change in width against $E_{0}$ cannot be observed in usual bulk carbon materials, in which the widths increase simply with increasing $E_{0}$ in our experience.
Sharpening of the edge image accompanies the transparentizing of the CM image. This transparency can be explained by the large increase in $n_{SE}$ relative to $n_{BE}$ with increasing $E_0$. On the other hand, from consideration of the SE images for $E_0$ ranging between 0.1-1.0 keV as described in Section 3.3, the edge effect cannot be related to SEs, BEs and EXSEs escaped from the volume $\Omega_{CM}$. The edge effect is possibly due to the probe electrons scattered from the copper plate under the CM, hereafter denoted as PESCs (probe electrons scattered from copper plate), with trajectories which could terminate in the copper plate.

The widths of the edge images can be evaluated from high magnification images of the images in Fig. 2. The brightness, i.e. the intensity, of the edge image can be described as a maximum at the extremal edge and decreasing rapidly to the background intensity of the CM image with increasing distance from the extremal edge $x$. The full width at half maximum intensity (FWHM) depends on the location of the edge image. Evaluated FWHMs are 25.1, 15.9, 14.0 and 12.5 nm for $E_0$ values of 2.0, 3.0, 5.0 and 7.0 keV, respectively, for examples. The FWHM levels off against $E_0$ for $E_0$ values at 7.0 keV and above. Assuming a Gaussian intensity distribution, the intensity distribution against $x$ can be depicted with functions $P(x)$ with $2.0 \text{ keV} \leq E_0 \leq 7.0 \text{ keV}$ as illustrated in Fig. 12, which is normalized so as to be $\int_{-\infty}^{\infty} P(x) dx = 1$. The half of the FWHM of $P(x)$ corresponds to the evaluated FWHM of the edge image. $P(x)$ can be set as $P(0) \approx P(x) \approx 0.00000$ (three significant figures) for $-3\text{FWHM} \leq x \leq 3\text{FWHM}$. The edge effect can possibly be interpreted phenomenologically as follows.

The function $P(x)$ in Fig. 12 can also be defined as that of PESCs in the interaction hemisphere, which could be directly backscattered from the copper plate in the column of the electron probe. Since the number of PESCs is so small that their total trajectory distribution in the interaction hemisphere is not affected by $P(x)$. $P(x)$ can be characterized as that for positive $x$ with each trajectory having a component along the scanning direction of the electron probe while for negative $x$ each trajectory has a component inverse to the scanning direction. With scanning of an electron probe, the interaction hemisphere, accordingly $P(x)$, moves along the scanning direction of the electron probe and passes through each of the edge surfaces near the specimen surface in succession as illustrated schematically in Fig. 13. Assuming a flat edge surface perpendicular to the $x$-direction, the carbon atoms in the edge are excited and release SEs to cause brightening up of the edge when a cross section of the hemisphere at rest is in the range of $x$, $0 \leq x \leq 3\text{FWHM}$, and overlaps with the edge surface. SEs continue to escape from the edge surface until the cross section for the peak of $P(x)$ overlaps. Accordingly the edge image can be seen to have a width. When the flat edge surface inclines from the perpendicular direction, i.e. the electron probe direction, to the $x$-direction, brightness of the edge could be diminished with increasing inclination angle at first and then could disappear.

4. Concluding Remarks

The secondary electron (SE) image of the surface of a thin carbon microgrid (CM) firmly covering a copper plate, the thickness of the CM being estimated to be 40 nm, was observed using a deceleration optics system SEM. Observation was made with the electron probe accelerated at voltages in a range of 0.1-30.0 keV. Characteristic SE images as a function of accelerating voltage $V_0$ and/or probe electron energy $E_0$ were obtained;

1) Clear SE images of the surface of the CM with $E_0$ below 1.0 keV

2) A drastic change of the CM texture of the SE image occurring at $E_0$ of 2.0 keV

3) Start of transparentizing CM texture and visualization of the image of the granular surface texture of the copper plate under the CM at $E_0$ of 2.0 keV

4) Improvement of transparentizing of the CM texture and clearer copper texture image with increasing $E_0$

5) Edge effect for the CM appearing at $E_0$ of 2.0 keV and above accompanying transparentizing of the CM textures

The $E_0$ value of 2.0 keV corresponds to the interaction hemisphere radius of 74 nm, accordingly the previously evaluated thickness of the CM of 40 nm is an overestimate and the thickness of the CM should be 37 nm or a little lower. The truest images of the CM surface can be obtained at $E_0$ values of 1.0 keV and below.
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References